En Chem, Inc.

Quality Assurance Document

SET No: 1

STANDARD OPERATING PROCEDURE

DEPARTMENT: Inorganic – Metals

INSTRUMENT TYPE: Inductively Coupled Plasma – Atomic Emission Spectroscopy

APPLICATION: Dissolved waters. Digested sample matrices including water, soils, biota, industrial wastes, sludges, sediments, solid wastes, and leaching extracts for the determination of metals.

REFERENCES: USEPA Test Methods for Evaluating Solid Wastes SW-846, 3rd Edition, Method 6010B Revision 2 (December 1996)
EPA Method 200.7 Rev. 4.4

PROCEDURE SUMMARY:

Prior to analysis, samples are digested using appropriate sample preparation method (See SW 846 Methods 3005A, 3010A, 3050B, 3015, and 3051). Samples are introduced by a peristaltic pump, nebulized, and the resulting aerosol introduced to the plasma torch. In the plasma, element specific atomic line emission spectra are produced. A grating disperses the spectra and the intensities of the lines are measured by photomultiplier tube. Background correction is required for trace metal analysis. The background points selected may be on either or both sides of the analyte line of interest and should be in an area free of spectral interference.

Detection Limits:
See Appendix 1.

Range of Measurement:
See Appendix 1.

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Annual Review

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DEFINITIONS:

Laboratory Water – eighteen mega-ohm water.

Calibration Blank (CAL BLK) – a volume of laboratory water containing five-percent nitric acid and five-percent hydrochloric acid volume/volume used in calibrating the instrument.

Calibration Standard – a solution prepared from single and/or mixed stock standards in laboratory water and acid used to calibrate the instrument.

Initial Calibration Verification (ICV) – a solution prepared in laboratory water and acid from a second independent source used to verify the calibration.

Initial Calibration Blank (ICB) – a solution prepared in laboratory water and acid used to validate the calibration.

Continuing Calibration Verification (CCV) – a solution prepared in laboratory water and acid used to verify the calibration.

Continuing Calibration Blank (CCB) - a solution prepared in laboratory water and acid used to validate the calibration.

Reagent Water – acidified laboratory water used as a diluent in the preparation of sample dilutions and serial dilutions.

Internal Standard – a fixed concentration standard used to adjust for variances in matrix and operating conditions.

Instrument Detection Limit (IDL) – the concentration equivalent to a signal, due to the analyte, which is equal to three times the average of the standard deviation of a series of seven replicates done on three non-consecutive days.

Method Detection Limit (MDL) – the concentration equivalent to a signal, due to analyte, which is equal to three times the standard deviation of a series of seven replicates.

Instrument Profile – the process of optimizing an analytical peak.

Linear Range (LR) – the concentration range over which the instrument response is linear.

Method Blank – a laboratory water that is treated in the same manner as a group of samples.

Laboratory Control Sample (LCS) – laboratory water spiked at known concentration and treated in the same manner as a group of samples.

Laboratory Control Sample Duplicate (LCSD) - laboratory water spiked at known concentration and treated in the same manner as a group of samples.

Matrix Spike (MS) – a sample spiked at known concentration and treated in the same manner as a group of samples to determine accuracy.

Matrix Spike Duplicate (MSD) - a sample spiked at known concentration and treated in the same manner as a group of samples to determine accuracy and precision.

Stock Standard – a single or mixed element standard purchased from vendors.

Duplicate (DUP) – a second aliquot of sample treated in the same manner as a group of samples used to determine precision.

Analytical Post Spike (PS) – a post-digestion spike performed on a sample in a digestion group.

Serial Dilution – a test performed on a sample in a digestion group or analytical batch comparing the sample result with a 1/5 dilution of the same sample.

Sensitivity Check (CRI) – a low level standard used to evaluate the calibration curve.

Interelement Correction Factor (IEC) – a factor used to compensate for spectral interferences.

Background Correction Point - an offline point selected to correct background.

Interference Check Sample (ICS) – a group of solutions containing both interferents and analyte elements of known concentration used to evaluate background and interelement correction factors.

Initial Demonstration of Capacity (IDC) – testing performed for each digestion method by every analyst.

Reporting Limit – a limit below which analytes are reported as not detected. Normally referred to as Estimated Quantitation Limit (EQL), MDL, or Project Specific Reporting Limit (PSRL).

SAMPLE HANDLING AND PREPARATION:

Aqueous samples are collected in plastic 250-mL, 500-mL, or 1000-mL bottles and preserved by adding HNO_3 to obtain a pH < 2. Once preserved, the sample holding time may not exceed 6 months. Solid samples may be collected in plastic or glass containers and refrigerated at 4°C.

Samples are prepared for ICP analysis using EPA analytical digestion methods. For recoverable metals see Method 3005A. For total metals see Method 3010A or 3015. For solid or biota samples see Method 3050Bor 3051. Dissolved metals do not need digestion if they were field filtered and acidified at the time of collection.

APPARATUS and EQUIPMENT:

Thermo-Jarrell Ash 61E Trace, Inductively Coupled Argon Plasma Spectrophotometer
50-mL, 100-mL, 200-mL and 500-mL class A volumetric flasks
Pipettes, adjustable and fixed
Liquid Argon – high purity
Liquid Nitrogen

INTERFERENCES:

Spectral interferences are caused by overlap of a spectral line from another element, background noise contribution, stray light from the line emission of high concentration elements and overlap from molecular band spectra. An interelement interference table (see current table) has been developed to help the analyst identify potential spectral interferences. Selecting background correction points adjacent to the analyte line can compensate for stray light and background noise contribution. Spectral overlaps can be compensated by equations that correct for interelement contributions.

Physical interferences are effects associated with sample introduction and nebulization. Changes in viscosity, especially in samples with high dissolved solids and high acid concentrations, can cause significant inaccuracies during analysis. Use of a peristaltic pump and an internal standard minimize these effects.

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with ICP. They can be reduced by matrix matching and standard additions if observed.

REAGENTS:

Hydrochloric Acid (HCL), trace metal grade.

Nitric Acid (HNO_3), trace metal grade.

Reagent Water – in a 500-mL volumetric flask add 200-mLs laboratory water, 25-mLs nitric acid, 25-mLs hydrochloric acid and bring to volume with laboratory water.

Yttrium Intermediate standard – in a 500-mL flask add 200-mLs laboratory water, 5-mLs nitric acid, 50-mLs 1000-ppm Yttrium stock standard and bring to volume with laboratory water.

Internal standard solution – in a 500-mL flask add 200-mLs laboratory water, 25-mLs nitric acid, 25-mLs hydrochloric acid, 25-mLs Yttrium Intermediate standard, 30-mLs 10,000-ppm Lithium standard and bring to volume with laboratory water.

STANDARDS:

GENERAL NOTES:

All standards less than 1 ppm are made daily.
Standards used for Calibration are made from stock standards purchased from High Purity.
Standards used to prepare ICV/CCV solutions are prepared using stock standards purchased for Inorganic Ventures.
All metals standards are prepared in class A volumetrics.
Standards are prepared using fixed and variable pipettors. Please refer to LAB-1 for the pipettor calibration procedure.
Standards are not used past the vendors' expiration date listed on the stock standard.
All prepared standards are recorded in the ICP Standard Logbook.

STOCK STANDARDS:

QCS-19 is purchased from High Purity. It contains 19 elements at 100-ppm each.

IV-19 is purchased from Inorganic Ventures. It contains 19 elements at 100-ppm each.

QCS-7 is purchased from High Purity. It contains 7 elements.

ICL500-6 is purchased from Environmental Express. It contains Al, Ca, and Mg at 5000-ppm, and Fe at 2000-ppm.

Single element stock standards are purchased in 1000-ppm and 10,000-ppm concentrations. All stock standards are logged into the Metals Stock Standard Logbook.

ANALYTICAL STANDARDS:

IV Calibration Standard – in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 2.0-mLs of QCS-7, 0.98-mLs 10,000-ppm sodium, and 0.30-mLs 10,000-ppm potassium. Bring to volume with laboratory water.

HP Calibration Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 10-mLs hydrochloric acid, and 2.0-mLs of QCS-19. Bring to volume with laboratory water.

STD3 Calibration Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, and 4.0-mLs each of 10,000-ppm single stock standards of calcium, aluminum, iron and magnesium. Bring to volume with laboratory water.

SNNA Calibration Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 0.2-mLs of 10,000-ppm tin, and 0.5-mLs 10,000-ppm sodium. Bring to volume with laboratory water.

MG Calibration Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, and 4.0-mLs of 10,000-ppm magnesium. Bring to volume with laboratory water.

ICVSP/CCVSP Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 1.0-mLs of IV-19, 0.2-mLs 10,000-ppm potassium, 1.98-mLs 10,000-ppm calcium and 1.98-mLs 10,000-ppm magnesium. Bring to volume with laboratory water.

ICVQC/CCVQC Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 0.1-mLs each of tin, barium, boron and silver; 2.0-mLs each of 10,000-ppm aluminum and iron; and 0.5-mLs 10,000-ppm sodium. Bring to volume with laboratory water.

Sensitivity Check (CRI-EQL) – in a 50-mL flask add 20-mLs laboratory water, 2.5-mLs nitric acid, 0.5-mL 1-ppm antimony, 0.5-mLs ICP CRI EQL intermediate, and 0.25-mLs 1-ppm silver. Bring to volume with laboratory water.

ICSA - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 10-mLs hydrochloric acid, and 20-mLs ICL500-6. Bring to volume with laboratory water.
ICSAB – in a 100-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 10-mLs hydrochloric acid 0.050-mLs each of 1000-ppm tin, nickel, cadmium and zinc; 10-mLs ICL500-6, 0.5-mLs IV-19 or QCS-19, and 0.5-mLs QCS-7. Bring to volume with laboratory water.

Solution C is used for analytical post spikes, and LCS, LCSD, MS and MSD spiked during sample preparation. The solution is verified before use. To test, dilute with reagent water and analyze as a sample.
In a 500-mL flask add 50-mLs of laboratory water, 5-mLs of nitric acid, and the following:

25-mLs 10,000-ppm sodium
23.75-mLs each of 10,000-ppm calcium and magnesium
12.5-mLs 10,000-ppm potassium
12.5-mLs each of 1000-ppm barium, boron and tin
1.25-mLs 10,000-ppm aluminum
1.25-mLs 1000-ppm silver
125-mLs 100-ppm QCS-19 or IV-19.

Bring to volume with laboratory water.

TCLP Spike Solution (TRTCP). This is used for spiking TCLP leachates. The solution is verified before use. To test, dilute with reagent water and analyze as a sample.
In a 200-mL flask add 30-mLs laboratory water, 2-mLs nitric acid 20-mLs each of 1000-ppm arsenic, lead, and chromium; 25-mLs 1000-ppm barium, 5.0-mLs each of 1000-ppm tin and boron; 0.5-mLs 1000-ppm silver, 0.5-mLs 10,000-ppm aluminum, and 50-mlS of IV-19 or QCS-19. Bring to volume with laboratory water.

INSTRUMENT OPERATION:

All ICP methods use a two-point calibration (a blank and a standard—see Appendix 2) for each analytical line. Calibration is verified prior to sample analysis by the use of Initial Calibration Verification (ICV) standard that is prepared from a source independent of the calibration standards. The instrument is calibrated after a thirty-minute warm-up and after profiling. Specific acceptance criteria for each QC standard are provided below. Details of instrument operation for profiling and calibration can be found in EN CHEM SOP 2-MET-42 "Inductively Coupled Plasma Emission Spectrometer", which is the Instrument Operating Procedure for the ICP.

SAMPLE ANALYSIS:

Samples are analyzed after the calibration is verified and the IEC corrections are checked. The calibration is verified by analyzing the ICV and ICB as noted in the QUALITY CONTROL section below. The ICSA and ICSAB solutions are analyzed to verify the IEC corrections factors.

NOTE: Any aqueous sample that is analyzed for selenium and is determined to have a detectable concentration present must be confirmed by GFAA. The only exception is TCLP extraction.

QUALITY CONTROL:

Initial Demonstration of Performance

Factory qualified service engineers install ICP's. The service engineer certifies that the instrument is functioning according to manufacturer's specifications prior to use by the laboratory. After certification by the service engineer, a qualified analyst prepares the analytical methods.

Aspirating a 1ppm Cd solution optimizes the nebulizer pressure. The signal to noise ratio is calculated at various pressures. The pressure that gives the best S/N ratio is entered into the analytical method for use.

Wavelength scans are performed on single element stock standards to determine where background correction points can be placed off line. TJA Trace ICP's have fixed background points for Se and Pb. After the background correction points are established, they are not changed.

Method Detection Limits (MDL)

MDLs are determined according to CFR 40 Pt.136, Appendix B. They are determined after initial installation, yearly thereafter, or whenever there are significant changes in analysts, operation or environmental conditions. Method Detection Limits are determined by preparing seven aliquots of laboratory water spiked at 3-5 times the estimated detection limit and processed through the entire digestion and analytical method. The instrument is calibrated and verified as in a normal analytical run.

Instrument Detection Limits (IDL)

IDLs are determined after initial installation, and quarterly thereafter. IDLs are determined by preparing seven aliquots of reagent water spiked at 3-5 times the estimated detection limit. The aliquots are analyzed on three non-consecutive days. The average of the three standard deviations is computed and multiplied by three to determine the IDL.

Linear Ranges

Linear ranges are determined after the instrument is calibrated and verified. Single element or mixed stock standards may be used. The upper range limit is an observed signal extrapolated from the calibration curve that is ± 10% of the true value. Linear ranges are verified at six-month intervals.

Interelement Correction Factors

Interelement Correction Factors are determined after setting background points and determining MDLs, EQLs, and Linear Ranges. Single element standards are analyzed after calibration and verification. The single element stock standards must be free of contamination or they will give false results. The single element standards are analyzed at a concentration at or below the Linear Range. Any signal greater that the absolute value of the EQL is subject to review as a possible interference. Interferences can be positive or negative depending on the interference. IEC's are adjusted as needed and reviewed every six months. See Appendix 3.

Initial Demonstration of Capability (IDC)

Each analyst for each digestion analyzes IDC solutions. The IDC solutions are carried through the entire digestion procedure. The results of the IDC's must be ± 20% of the true value and are kept on file for each analyst.

<u>Daily Quality Control</u>

Initial Calibration Verification (ICV)

The ICV is prepared from a source independent from the calibration standards and must be analyzed immediately after calibration. The ICV must meet the rejection criteria of ± 10% of the true value. Also, the relative standard deviation must be < 5 % from replicate (minimum of two) integrations. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-point of the calibration curve. For EPA Method 200.7, the ICV must meet the rejection criteria of ± 5% of the true value.

Initial Calibration Blank (ICB)

The ICB must be analyzed after the ICV. The absolute value of the ICB must be ≤ 3 x IDL. If 3 times the IDL is less than 1/10 the EQL, 1/10 the EQL is used as the control limit. Also, if three times the IDL exceeds the EQL, the EQL is used as the control limit. In cases where 3 x the IDL criteria are consistently exceeded, the QC officer may determine a limit from historical data. Recalibrate if the ICB fails. However, if the ICB concentration is < 1/10 the concentration of the associated samples, the analysis need not be terminated. Current control limits for ICB are established quarterly and maintained on file in the laboratory.

Sensitivity Check (CRI)

The sensitivity check is normally run after the ICB. The concentration of the CRI will usually be at the EQL. Pass/fail criteria and CRI concentrations may vary due to client request.

Continuing Calibration Verification (CCV)

The CCV is analyzed after every 10 samples. Rejection criteria is ± 10 % of true value. The relative standard deviation must be < 5% from replicate (minimum of two) integrations. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve. For ICP analysis, if the Caves that bracket the samples to be reported for the analytes of interest are within the acceptable limit, those analytes may be reported.

Continuing Calibration Blank (CCB)

The CCB is analyzed after every CCV. The absolute value of the CCB should be ≤ 3 x IDL. If 3 times the IDL is less than 1/10 the EQL, 1/10 the EQL is used as the control limit. Also, if three times the IDL exceeds the EQL, the EQL is used as the control limit. In cases where 3 x the IDL criteria are consistently exceeded, the QC officer may determine a limit from historical data. Recalibrate if the CCB fails. However, if the CCB is < 1/10 the concentration of the associated samples, analysis need not be terminated. Current control limits for CCB are established quarterly and maintained on file in the laboratory.

Laboratory Control Sample (LCS/LCSD)

The LCS is carried through all preparation procedures and analyzed for each batch of 20 or fewer samples. See current QC Charts for control ranges. In cases where the LCS or LCSD is outside of acceptable ranges all samples prepared in that batch must be redigested and/or reanalyzed. In situations where the LCS or LCSD fails, and redigestion is not possible, the "**&**" flag is applied to the LCS, LCSD and ALL associated samples including method blanks, MS and MSD. LCS control limits are updated yearly based on historical data for each digestion method.

Method Blank (MB)

A MB is carried through the digestion procedure and analyzed with each batch of 20 or fewer samples. Rejection criteria for the MB are dependent upon the specific reporting limits for a project and any applicable regulatory limits. Appendix 4 to this SOP includes specific instructions for rejection of the MB or qualification of the associated data in accordance with various reporting limits.

Serial Dilution Test

A Serial Dilution Test is performed on each digestion group (batch). For dissolved samples that are not digested, one serial dilution is done per twenty samples. A 1/5 dilution is analyzed and compared to the original sample result. The serial dilution times 5 should agree within ± 10% of the original value. If the results do not agree, the sample is flagged with an "E". The sample is not qualified if the result is less than 50X the IDL. The serial dilution is used to determine if matrix interferences are present. It is left to the analyst to determine if additional corrective action is required.

Analytical Post Spike

An Analytical Post Spike is required at a frequency of 5 % or per sample digestion group. The control limits for a post-spike are 75-125%. If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample, or use a more appropriate concentration if necessary. Dilute appropriately until an acceptable recovery is obtained. For EPA Method 200.7, the post-spike recovery must be within 85-115%.

ICSA / ICSAB

Analytes present in the ICSA and ICSAB solutions should be recovered within 80-120% of the true value. There is no criteria established for elements not present in the ICSA solution. However, if the result of an element not present in the solution is greater than ± EQL, investigate whether a problem exists. A concentration greater than ± EQL can indicate contamination, poor replicates, or an improperly adjusted IEC.

Trace elements such as Cd, Se, Pb, Sb, and Tl are the most likely to be out of control. The ICSA is composed of 500-ppm AL, Ca, Mg, and 200-ppm Fe. If the element has 2 or more correction factors both individual solutions should be analyzed to determine which is the source of the QC failure.

> Example: ICSA produces a Pb result of 5.7 μg/L. The EQL is 5.0 μg/L. Al and Fe are both interferents. Run a 500-ppm Al and a 200-ppm Fe solution to determine if one or both of the IECs needs to be adjusted. Adjust IEC as per IEC appendix. If individual solutions are in control, sample analysis may be started. Analyses of IEC test solutions are counted as samples when determining the number of samples between the ICV and CCV.

Linear Range (LR)

Any analyte or interfering element greater than the linear range must be diluted until it is less than the linear range. Any analytes that have interfering elements greater than the linear range require that the interfering element be within the linear range for the reported analyte to be valid.

For EPA Method 200.7, analytes that are within 10% of the linear range must be diluted.

Internal Standard

The internal standard counts for a given analysis must be within 30-120% of the internal standard counts of the Calibration Blank.

Accuracy

One matrix spike and matrix spike duplicate are digested and analyzed for each group of 20 or fewer samples that are similar in matrix at a frequency of 5%. For EPA Method 200.7, a matrix spike and matrix spike duplicate are performed at a frequency of 10% or one per sample batch, whichever is more frequent. Both QC samples must be calculated for accuracy. See current QC charts for control range.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If one or both spike recoveries are outside of the specified control limit, the corresponding parent sample and MS/MSD are to be flagged with an "N" qualifier. An analytical post spike is required if either the MS or MSD fail for accuracy.

If the analyte of interest is greater than 4 times the level of the spike concentration, accuracy calculations are not necessary and the "N" flag need not be applied.

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and LCSD must be used in its place.

Precision

Matrix spike duplicate samples (MSD) or duplicates (DUP) are analyzed at a frequency of 5% or one per batch of samples that are similar in matrix. For EPA Method 200.7, a matrix spike duplicate of sample duplicate is performed at a frequency of 10% or one per sample batch, whichever is more frequent.

For precision, relative percent difference (RPD) is used to calculate compliance. See current QC charts for control limits.

Calculation:

$$RPD = \frac{MS - MSD}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with a "*" flag.

For duplicate samples that are less than 5 times the EQL, use +/- the EQL for acceptance limits.

SAMPLE RESULT CALCULATIONS:

Aqueous Sample Calculation:

Raw Data result (µg/L) X DF X MW DF(if applicable) = Final Result (µg/L)

where: MW DF = microwave dilution factor or 1.11 (correction for 45 mL initial volume to 50 mL final volume)
DF = dilution factor

Soil Sample Calculation:

$$\frac{\text{Raw Data result } (\mu g/L) \text{ X FV (L) X DF}}{\text{Sample Weight (g) X Dry Weight (decimal form)}} = \text{Final Result (mg/kg dry weight corrected)}$$

where: DF = Dilution Factor
FV = Final Volume

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and the Hazardous Waste Contingency Plan.

APPENDIX 1

Method Analytes and Reporting Limits

Analyte	Wavelength	EQL Water µg/L	EQL Soil/Biota mg/Kg	EQL TCLP µg/L	Linear Range
Silver	328.0	5.0	0.50	50	10,000
Aluminum	308.2	200	20	200	50,000
Arsenic	189.0	10	1.0	200	500,000
Boron	249.6	100	10	100	10,000
Barium	493.4	5.0	0.5	200	10,000
Beryllium	313.0	1.0	0.10	50	10,000
Calcium	317.9	100	50	1000	500,000
Cadmium	226.5	1.0	0.10	50	50,000
Chromium	267.7	3.0	0.30	50	50,000
Copper	324.7	10	2.0	50	50,000
Cobalt	228.6	3.0	0.50	50	50,000
Iron	259.9	50	10	100	50,000
Iron	271.4	100	10	100	50,000
Potassium	766.4	100	100/500	1000	50,000
Magnesium	279.0	30	30/50	1000	500,000
Manganese	257.6	2.0	0.20/0.50	50	30,000
Molybdenum	202.0	10	1.0	50	50,000
Sodium	330.2	500	200/1000	1000	200,000
Nickel	231.6	5.0	0.50	50	50,000
Lead	220.3	5.0	0.50	200	50,000
Antimony	206.8	10	1.0	50	50,000
Selenium	196.0	10	1.0	200	50,000
Thallium	190.8	10	1.0	50	50,000
Tin	189.9	20	5.0	50	50,000
Titanium	334.9	20	2.0	50	50,000
Vanadium	292.4	5.0	0.50	50	50,000
Zinc	206.2	20	2.0/5.0	200	50,000

** Control limits and Linear Ranges are updated periodically. Those that are in use at the time of analysis will be used and made available to data validators.

APPENDIX 2

ICP Standard Components and Concentrations, µg/L

Element	ICVSP/ CCVSP	ICVQC/ CCVQC	ICSA	ICSB (AB)	Cal Std	Cal Std Name	Linear Range (+/- 10)	Spike **
Ag		500		500	1000	IV	10000	100
As	500			500	1000	HP	50000	1000
Al		100000	500000	500000	200000	STD3	500000	1000
B		500		500	1000	IV	30000	1000
Ba		500		500	1000	IV	10000	1000
Be	500			500	1000	HP	10000	1000
Ca	100000		500000	500000	200000	STD3	500000	20000
Cd	500			1000	1000	HP	50000	1000
Co	500			500	1000	HP	50000	1000
Cr	500			500	1000	HP	50000	1000
Cu	500			500	1000	HP	50000	1000
Fe259	500		200000	200000	1000	HP	50000	1000
Fe271		100000	200000	200000	200000	STD3	500000	1000
K404		100000			200000	STD3	500000	10000
K766	10000				25000	IV	50000	10000
Mg	100000		500000	500000	200000	STD3	500000	20000
Mn	500			500	1000	HP	20000	1000
Mo	500			500	1000	HP	50000	1000
Na330		25000			50000	IV	200000	20000
Na588	10000				25000	SnNa	50000	20000
Ni	500			1000	1000	HP	50000	1000
Sb	500			500	1000	HP	50000	1000
Sn		500		500	1000	SnNa	50000	1000
Pb	500			500	1000	HP	50000	1000
Se	500			500	1000	HP	50000	1000
Tl	500			500	1000	HP	50000	1000
Ti	500			500	1000	HP	50000	1000
V	500			500	1000	HP	50000	1000
Zn	500			1000	1000	HP	50000	1000

** Spike level for LCS, MS, MSD. Also post spike level when solution C is used.

APPENDIX 3

Interelement Correction Factor Set-up for TJA ICAP61E Trace

Purpose:

The interelement Correction Factors (IEC's) are used to correct for false signals (positive and negative) produced when peaks overlap in the spectra.

Initial Setting of the IEC's:

B. Single element standards are run with background correction points in place. The standards are run at or below the Linear Range. Any signal greater than the absolute value of the EQL should be evaluated as a possible interference. Wavelength scans are useful in determining if contamination is present. A copy of the MIT Wavelength Tables is available for analyst reference.

C. Use Part A for all elements other than Se and Pb.

A. Elements with results above the detection limits are noted. The following calculation is performed:

$$\frac{\text{Element Result } (\mu g/L)}{\text{Interfering Element Result } (\mu g/L} = \text{IEC}$$

This ratio is entered in the IEC table in the method file.

B. Pb and Se have two analytical lines, which are combined to produce the analytical result. Each Pb and Se line must be dealt with separately. Ratios will be entered for both the 220/1 and 220/2 in the case of Pb.
Example: A 500-ppm Al standard produces a –171 µg/L on the Pb 220/1 line and a 58 µg/L result on the Pb 220/2 line. The Al reads 497,100 µg/L. Using the above calculation we get –0.000344 for the Pb 220/1 and 0.000117 for the Pb 220/2.

Entering the IEC's:

B. At the main menu select **Development** and press enter. Select **Methods** and press enter. At the Enter Method Name prompt; type the method name or select it from the list. Depress the enter key. Press **F5 (Element Info)** and select **F3 (IEC's).** Move the cursor to the element you wish to enter an IEC for and press the *page down* key to access the wavelength of choice. Pressing the arrows keys will highlight the second wavelength if one is listed. Press the *page down* key to enter the IEC.

C. If the interfering element is not selected (listed) press **F6** (*edit*) and the **F6** (select IEC's) key. Choose the interfering element by entering the number corresponding to the element from the list. Press the enter key. Press **F9** (*done/keep*).

D. Then type the ratio under the k1 header in the appropriate row. Enter a negative before the ratio if it is a negative interference. Press **F9** (*done/keep*) as many times as necessary to return to the main menu and save the IEC's.

APPENDIX 4

Method Blank Qualification
1 of 3

Flowchart A
LOD/LOQ BLANK FLAGGING and REPORTING

	-RL		Zero		RL
5	4	3	1	2	6
		-LOD		LOD	

BLANK POSITION> / Sample Position⇓	5	4	3	1	2	6
1	R	A(-x) Flag Report < LOD	Report < LOD	Report < LOD	Report < LOD	Report < LOD
2	R	A(-x) Flag Report result w/20X Rule	Report result	Report result	A(x) Flag Report result w/20X Rule	R
3	R	A(-x) Flag Report <LOD	Report <LOD	Report <LOD	Report <LOD	Report <LOD
4	R	A(-x) Flag Report <LOD	Report <LOD	Report <LOD	A(x) Flag Report result w/20X Rule	Report <LOD
5	R	R	R	R	R	R
6	R	A(-x) Flag Report result w/20X Rule	Report result	Report result	A(x) Flag Report result w/20X Rule	Report result w/20X Rule -or- R if result is not 20XMB

R= Analyst discretion to Re-run,Re-calculate, or Re-digest
X= Value in Method Blank (MB).

APPENDIX 4

Method Blank Qualification
2 of 3

Flowchart B
EQL or PRSL BLANK FLAGGING and REPORTING

-RL **Zero** **RL**

5 4 3 1 2 6

-LOD **LOD**

BLANK POSITION> / **Sample Position** ⇓	5	4	3	1	2	6
1	R	Report < RL	Report < RL	Report < LOD	Report < RL	Report < RL
2	R	Report < RL	Report < RL	Report result	Report < RL	Report < RL
3	R	Report < RL	Report < RL	Report <LOD	Report < RL	Report < RL
4	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
5	R	R	R	R	R	R
6	R	Report result	Report result	Report result	Report result	Report result w/20X Rule -or- R if result is not 20XMB

R= Analyst discretion to Re-run, Re-calculate or Re-digest
X= Value in Method Blank (MB)
RL= Reporting Limit

Appendix 4

Method Blank Qualification
3 of 3

Flowchart C
EQL or PRSL BLANK ''B'' FLAGGING and REPORTING

	-RL			Zero		RL	
5		4	3		1	2	6
		-LOD			LOD		

BLANK POSITION> / **Sample Position⇓**	5	4	3	1	2	6
1	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
2	R	Report Result B - Flag A(-x) Flag W/ 20X Rule	Report Result B - Flag	Report Result B - Flag	Report Result A(x) Flag W/ 20X Rule	R
3	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
4	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
5	R	R	R	R	R	R
6	R	Report result A(-x) Flag W/ 20X Rule	Report result	Report result	Report Result A(x) Flag W/ 20X Rule	Report result A(x) Flag w/20X Rule -or- R if result is not 20XMB

R= Analyst discretion to Re-run,Re-calculate, or Re-digest
X= Value in Method Blank (MB).
RL= Reporting Limit